

# Bond Strength of 5<sup>th</sup>, 6<sup>th</sup> and 7<sup>th</sup> Generation Bonding Agents to Intracanal Dentin of Primary Teeth

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## Abstract

**Objectives:** This in-vitro study sought to assess the push-out bond strength of a total etch and 2 self-etch bonding systems to intracanal dentin of primary anterior teeth (PAT).

**Materials and Methods:** Thirty-six primary anterior teeth were randomly divided into 3 groups of 5<sup>th</sup> generation (Single Bond 2), 6<sup>th</sup> generation (Clearfil SE) and 7<sup>th</sup> generation (Single Bond Universal) bonding agents. The canal orifice was restored with composite resin and the push-out test was carried out to assess the bond strength. After applying the push-out load, specimens were evaluated under a light microscope at 40X magnification. One-way ANOVA and log-rank test on Kaplan-Meier curves were applied for the comparison of bond strength among the 3 groups.

**Results:** The mean± standard deviation (SD) bond strength was 13.6±5.33 MPa for Single Bond 2, 13.85±5.86 MPa for Clearfil SE and 12.28±5.24 MPa for Single Bond Universal. The differences in bond strength among the 3 groups were not statistically significant ( $P>0.05$ ).

**Conclusion:** All three bonding agents are recommended for use with composite posts in PAT. However, due to high technical sensitivity of the Total Etch system, single or two-step self etch systems may be preferred for uncooperative children.

**Key words:** Bonding agents; Bond strength; Primary teeth

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## INTRODUCTION

Early childhood caries are common in children and quickly lead to the loss of tooth structure in primary maxillary anterior teeth. Removal of carious tissue in these teeth renders pulp treatments inevitable and due to the lack of sufficient enamel, retention must be gained from the coronal 3 mm of the canal to restore the tooth crown [1]. The same protocol is applied and followed for dentin bonding in per-

manent and primary teeth [2]. However, in-vitro studies have reported controversial results regarding the bond strength of adhesive systems to primary compared to permanent dentin. Different characteristics of primary dentin may be responsible for the conflicting results reported in the literature [2, 3]. Greater thickness of peritubular dentin with higher percentage of mineralization and larger diameter of dentinal tubules in primary teeth [2] sig-

nificantly decrease the amount of solid dentin available for bonding compared to that in permanent teeth [2- 4]. On the other hand, different techniques have been used for the reconstruction of severely damaged primary anterior teeth; composite resin posts are most commonly used for this purpose [5].

Increased demand for esthetic restorations in dentistry has led to the development of different systems to enable adequate bonding to enamel and dentin with fewer steps [6]. Available dentin adhesives include 3-step, 2-step and one-step adhesives depending on the method of incorporation of the three main constituents of etching, priming and bonding [7]. Many researchers have attempted to improve the efficacy of dentin bonding agents. Now that adhesives have reached an acceptable level of bond strength, attempts have focused on reducing the application steps since the use of multi-step agents in children is difficult and time-consuming. Simultaneous enamel and dentin etching systems by using 2-step 5th generation bonding agents have shown favorable clinical efficacy. A recent revolutionary advancement in dentin bonding agents is the use of acidic adhesives enabling simultaneous application of acid, primer and bonding agent all together in 6th and 7th generation bonding systems [8]. Aside from the easy steps, the mechanism of action of 6th and 7th generation bonding agents is surface demineralization of dentin and simultaneous penetration of monomers into the resultant porosities [9]. One-step systems simplify and shorten the process of bonding and are beneficial for use in uncooperative children [3].

Considering the fact that intracanal dentin has significant structural differences with coronal dentin in terms of the number and diameter of dentinal tubules and the amount of peritubular dentin (dentinal tubules in the root are straighter, less divergent and not as numerous as in the crown)[10], materials and methods that compensate for the afore-mentioned limitations and provide maximum retention can

ensure greater durability of composite restorations in primary teeth. Thus, this study sought to assess and compare the push-out bond strength of three 5th, 6th and 7th generation bonding agents to intracanal dentin in PAT.

## MATERIALS AND METHODS

This experimental study evaluated 36 primary anterior teeth extracted due to severe caries in the past 6 months and stored in 0.5% Chloramine T solution for one week. The teeth were then stored in distilled water. Teeth crowns were cut 1mm above the cemento-enamel junction using diamond discs perpendicular to the long axis of the teeth. According to Torres et al, [13] and using the multiple means comparison feature, the minimum sample size for each of the 3 groups was calculated to be 12 teeth via Minitab software taking into account  $\alpha=0.05$ ,  $\beta=0.2$ , mean difference=3.3 and SD=3.16. A total of 36 teeth were randomly divided into 3 groups (n=12). The teeth in each group were coded in such a way to ensure blindness of results.

A 5th generation bonding agent (Single Bond 2)(3M, St. Paul, MN, USA), a 6th generation bonding agent (Clearfil SE) (Kuraray Co., Osaka, Japan) and a 7th generation bonding agent (Single Bond Universal)(3M, St. Paul, MN, USA) were used in groups 1, 2 and 3, respectively according to the manufacturer's instructions (Table 1).

Root canals were prepared using K files (Mani Inc.) up to #3, irrigated with saline solution and dried with paper points (PT Dent, USA) by the researcher (post graduate student of pediatric dentistry). The coronal 3mm of the canals was restored with a posterior composite resin (FiltekP60, 3M ESPE, USA). Composite resin was applied incrementally and each layer was light-cured for 20s using a light-curing unit. The composite was packed by a composite condensing instrument in such a way that increments obtained adequate C factor (the ratio of bonded to unbonded surfaces) (Figure 1).

**Table 1.** The understudy bonding agents and their composition

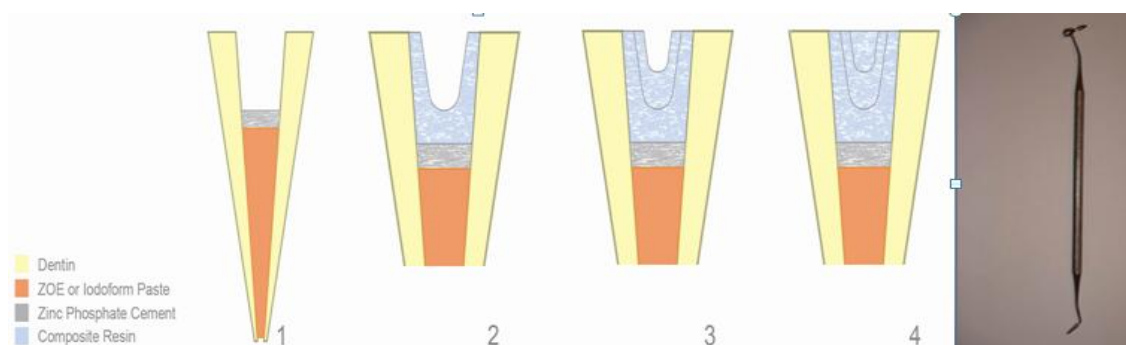
Materials	Components	Mode/steps of application	Manufacturer
Single Bond 2 Total-etch Self-priming	Dimethacrylate resins, HEMA, Vitrebond™ Copolymer, Filler, Ethanol Water, Initiators	15s of etching 10s of rinsing Drying with paper points Application of 2-3 coats of bonding agent 10s of curing	3M, St. Paul, MN, USA
Clearfil SE Bond Self-etch 2-steps	Primer: MDP, HEMA, Hydrophilic dimethacrylate, N,N-Diethanol p-toluidine, water  Bonding: MDP, Bis-GMA, HEMA Hydrophobic dimethacrylate, dl-Comphorquinone, N,N-Diethanol p-toluidine, silanized silicate	20s of priming Gentle air drying Application of bonding agent Gentle air drying 10s of curing	Kuraray Co., Osaka, Japan
Single Bond Universal Self-etch 1-step	MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond™ Copolymer, Filler, Ethanol, Water, Initiators, Silane	Application of adhesive to the tooth surface for 20s 5s of gentle air drying 10s of curing	3M, St. Paul, MN, USA

**Table 2.** The mean bond strength (MPa) and SD values in the primary teeth bonded with 5<sup>th</sup>, 6<sup>th</sup> and 7<sup>th</sup> generation bonding agents using one-way ANOVA and log-rank test on Kaplan-Meier curves with consideration of failure modes

Statistical test	Single Bond 2 (5 <sup>th</sup> generation)	Clearfil SE bond (6 <sup>th</sup> generation)	Single Bond Universal (7 <sup>th</sup> generation)
One-way ANOVA	13.6(±5.33)	13.85(±5.86)	12.28(±5.24)
Kaplan-Meier with consideration of failure modes	17.24(±5.33)	13.85(±1.89)	13.17(±1.56)

**Table 3.** The frequency percentage of bond failure modes of intracanal dentin of primary anterior teeth using 5<sup>th</sup>, 6<sup>th</sup> and 7<sup>th</sup> generation bonding agents

Type of bonding		Failure modes			
		Adhesive	Mixed	Cohesive in dentin	Cohesive in composite
Fifth generation	Number	2	4	1	6
	Percentage	15.4%	30.8%	7.7%	46.2%
Sixth generation	Number	0	12	0	0
	Percentage	0.0%	100.0%	0.0%	0.0%
Seventh generation	Number	1	10	0	2
	Percentage	7.7%	76.9%	0.0%	15.4%



**Fig1.** The composite was packed by a composite condensing instrument in such a way that increments obtained adequate C factor

All specimens were cured under similar conditions using a Halogen light-curing unit (Dentus, Italy) with an intensity of 350 mw/cm<sup>2</sup> and 2mm distance from the tip to the teeth. The teeth were then mounted in transparent acrylic blocks. Specimens were stored in distilled water at 37°C until sectioning and bond strength testing. A 1mm thick section was made in each tooth at the prepared site using a water-cooled diamond blade on Mecatome cutting machine (Perci, T201A, France)

The push-out shear bond strength test was performed using Zwick/Roell Z05 universal testing machine (Ulm, Germany). The load was applied to the respective area in an apico-cervical direction with a crosshead speed of 0.5 mm/min via a stainless steel cylindrical plunger with a diameter corresponding to that of the canal. The highest load applied causing debonding was recorded in Newtons (N). To report the bond strength in Megapascals (MPa), the recorded load in N was divided by the respective cross-sectional area (mm<sup>2</sup>). Thus, before the push-out test, both sides of each section were photographed by a digital camera (Canon, Eos600D, Japan) and images were entered into Auto CAD software (version 2013). The cross-sectional area was calculated using  $(A1 + A2) h/2$  equation.

After applying the push-out load, specimens were evaluated under a light microscope (Olympus, Sxz2-zb16, Japan) at 40X magnification to determine the mode of failure.

The mode of failure was categorized as cohesive, mixed or adhesive. Data were analyzed using ANOVA and log-rank test on Kaplan-Meier curves. In all statistical tests,  $P \leq 0.05$  was considered statistically significant.

## RESULTS

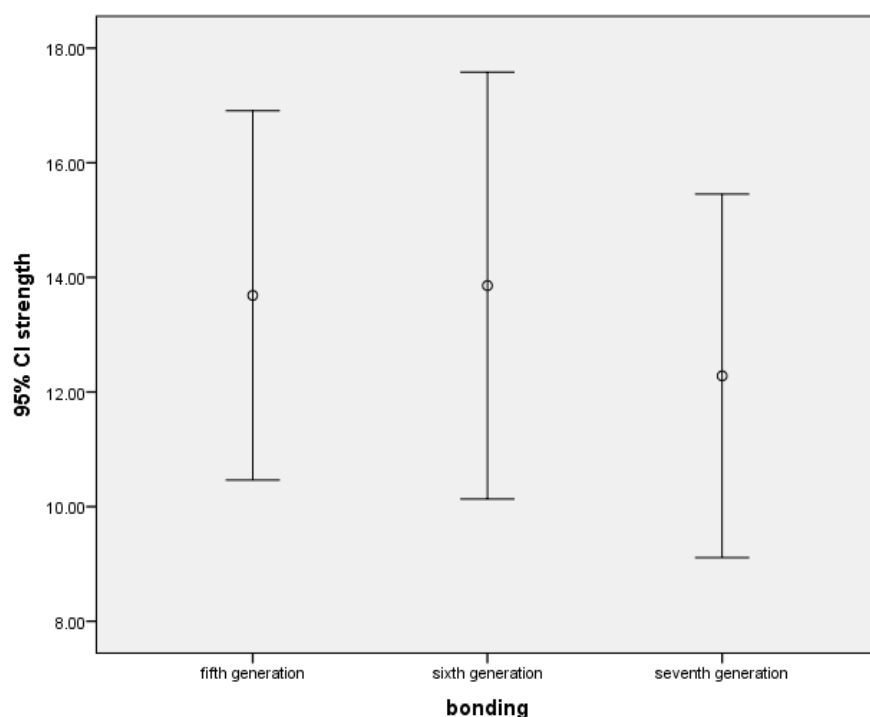
Analysis of data with one-way ANOVA revealed that the mean bond strength of understudy bonding systems was not significantly different ( $P=0.73$ ). Analysis of data with log-rank test on Kaplan-Meier curves with consideration of the failure mode also showed that the mean bond strength was not significantly different among Single Bond Universal, Clearfil SE or Single Bond 2 ( $P=0.218$ , Table 2) (Diagrams 1 and 2).

The frequency percentage of failure modes is shown in Table 3.

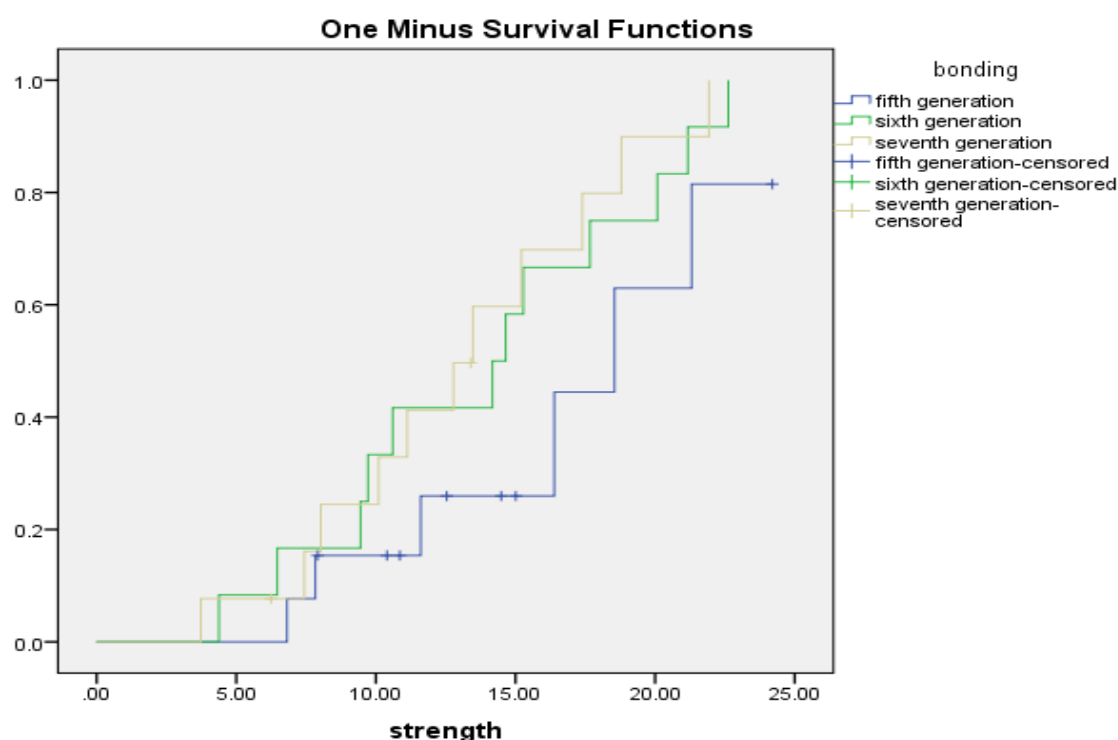
Evaluation of failure mode by Fisher's exact test revealed a significant difference between 5th and 6th generation bonding agents ( $P=0.005$ ) but failed to find a statistically significant difference between the 5th and 7th ( $P=0.11$ ) or 6th and 7th ( $P=0.2$ ) generation bonding agents.

## DISCUSSION

Clinical success of composite restorations depends on the adhesive system and its ability to achieve a strong composite-dentin bond [8]. The push-out test exerts a shear load on the bonding agent-composite and bonding agent-



**Diagram 1.** Error bar of mean and 95% confidence interval of composite bond strength to intracanal dentin in primary anterior teeth using 5<sup>th</sup>, 6<sup>th</sup> and 7<sup>th</sup> generation bonding agents



**Diagram 2.** The cumulative frequency distribution of composite bond failure to intracanal dentin in primary anterior teeth with 5<sup>th</sup>, 6<sup>th</sup> and 7<sup>th</sup> generation bonding agents using log-rank test on Kaplan-Meier curves with consideration of failure mode

dentin interfaces. Push-out shear test is more similar to the clinical setting than the linear shear test [11].

Several factors affect the bond strength to dentin under in-vitro conditions namely the type and age of tooth, degree of dentin mineralization, bonded dentin surface, type of bond strength test (shear or tensile), storage media of teeth, relative humidity of the environment in the substrate and testing conditions.

These variables are responsible for the wide variability of bond strength data reported in the literature [8].

Morphological differences exist between the bonds of total etch and self-etch systems. One difference is in the thickness of the hybrid layer [12].

The mechanism of dentin bonding is based on the formation of resin-dentin interface at the inter-diffusion zone [13].

Although the hybrid layer created by the total etch systems is thicker than that formed by the self-etch systems, comparison of bond strength between the two has yielded controversial results [12]. Hybrid layer thickness does not play a key role in success and dentin bond strength is probably proportionate to the resin tags interlocking with collagen fibers as well as the quality of the hybrid layer [14]. The resin tags formed with the etch-and-rinse adhesives are much longer than those found in self-etching adhesives but both systems form a continuous and uniform hybrid layer (in terms of thickness) [12].

In our study, all three bonding systems of Single Bond 2, Clearfil SE bond and Single Bond Universal had acceptable bond strength to intracanal dentin of primary anterior teeth and no significant difference was found among the bond strength values.

To the best of our knowledge, there is no similar study evaluating the push-out bond strength of different bonding systems to intracanal dentin of primary anterior teeth.

However, several studies have evaluated the bond strength (shear and tensile) to dentin

close to the dentino-enamel junction of primary teeth. Yaseen et al. [8] compared the shear bond strength of two self-etch systems (6th and 7th generations) and Senawongse et al. [15] measured the microshear bond strength of total etch (Single Bond) and self-etch (Clearfil SE Bond) adhesives to primary dentin. The bond strength value obtained in the aforementioned studies was greater than our rate; but similar to our study, they failed to find a significant difference between different bonding systems. The higher shear bond strength reported in their studies may be attributed to morphological differences of the bonded dentin at different areas of the tooth and also difference in size of the cross-sectional area where the load is applied.

The above-mentioned studies evaluated the shear bond strength to dentin away from the pulp (dentin close to the dentino-enamel junction) that has fewer dentinal tubules with smaller diameters than dentin around the pulp, which contains more dentinal tubules with larger diameters. Thus, dentin away from the pulp is more calcified and therefore is a better substrate for etching and bond formation leading to higher bond strength. Also, another difference between the mentioned studies and ours is in the cross sectional area where the load was applied; they evaluated shear bond strength and load applied to a smaller area compared to our study.

Previous studies have shown that the bond strength is influenced by three factors namely pH, solvent properties and filler content of the adhesive [6].

### **Effect of pH:**

In the total etch system, primer and adhesive are mixed in one bottle; which is applied to the surface after etching with 37% phosphoric acid.

These bonding systems create mechanical retention with the etched dentin by forming resin tags and hybrid layer [6]. Single Bond 2 belongs to this group.

Self-etch systems are classified into 3 groups of mild, intermediate-strong and strong based on their ability to dissolve the smear layer and demineralize the subsurface dentin.

The pH in the strong self-etch systems is equal or less than one and their bonding mechanism is similar to that of total etch systems; whereas, the pH is between 1 and 2 in intermediate-strong self-etch systems and  $\geq 2$  in mild self-etch systems [14, 16].

Adhesive systems with a very low pH cause very deep etched surfaces and since the etchant in total etch is separate from the bonding agent, it can lead to discrepancy between the degree of etching and dentin demineralization with the penetration of bonding agent. In this situation, deep layers of decalcified dentin are not completely saturated with the adhesive agent. The unsaturated etched space remains as a mechanically weak area and causes nano-leakage and decreased bond strength [8,14]. However, in the self-etch systems, depth of dentin demineralization and resin monomer penetration is equal and demineralization and resin penetration occur simultaneously [3]. Therefore, in the total etch system we may expect greater variability in retention compared to self-etch systems; variable modes of failures in the results of total etch system can be somehow related to this characteristic.

Considering the fact that acid penetration primarily occurs along the tubules, presence of dentinal tubules with greater diameter in primary dentin can lead to deeper penetration of acidic conditioner and subsequently stronger demineralization [9].

Acids used for dentin surface conditioning eliminate the smear layer in primary teeth much faster than in permanent teeth. Thus, shorter conditioning time or using a weak acidic solution is recommended for primary teeth. Studies have shown that shorter conditioning time in primary teeth not only dissolves the smear layer, but also creates a surface morphology similar to that of conditioned permanent dentin [8].

### ***Effect of solvent:***

The solvents used are primarily water. Some bonding agents may have acetone or ethanol. Resultantly, primers have different vaporization speed, drying patterns and penetration properties that can all influence the bond strength [14]. It has been reported that water-based bonding systems due to the incomplete polymerization of monomer cause lower bond strength [9, 13]. It has been reported that bonding systems containing acetone solvent can better react with the substrate due to the higher volatility of acetone compared to ethanol [6, 13]. Since the solvents of all bonding systems evaluated in our study were water- and ethanol-based, it does not seem to have affected the results.

### ***Effect of filler:***

Several studies have confirmed the effectiveness of filled bonding agents in resin bond strength [6,17]. Based on the literature, application of filled bonding agents to the tooth surface is easier and they provide higher in-vitro bond strength [5,14]. It has been reported that presence of 10% filler in the adhesive is necessary to increase bond strength [17]. However, some studies did not find any significant difference in the bond strength of filled and unfilled bonding systems [6].

In our study, all three bonding systems evaluated were filled; which may have affected the bond strength values.

Both understudy self-etch systems contained 10-MDP (methacryloxy decyl dihydrogen phosphate) hydrophilic acidic monomer. It has a molecular structure capable of chemically reacting with hydroxy apatite remnants following acid etching. The produced chemical salt has hydrophilic stability [17]. Several studies have attributed the high bond strength of Clearfil SE to the presence of this monomer in its composition [3, 6, 8, 12, 17]. Presence of MDP in self-etch systems in this study may be responsible for their comparable bond strength to that of total etch systems.

**Assessment of push-out bond strength:**

Numerous studies are available regarding the push-out bond strength to intracanal dentin in permanent teeth with controversial results. In a study by Alizadeoskoei et al, on the push-out bond strength of fiber-reinforced composite posts to intracanal permanent dentin with the use of different adhesive systems, they concluded that the bond strength of total etch systems is higher than that of one- and two-step self-etch systems; they stated that in etch and rinse systems by the use of phosphoric acid and etchants the smear layer is completely dissolved creating a path for hybridization of decalcified intertubular dentin, dentinal tubule walls and accessory canals. Self-etch primers have weak etching ability and cannot completely eliminate the smear layer and only partial penetration through the smear layer may be seen [18]. Alizadeoskoei et al, first prepared a post space in the canal by drilling and then restored the canal and sectioned the root into 3mm thick slices. The push-out bond strength reported in their study was greater than our obtained value; which may be due to the anatomic differences between primary and permanent dentin and the reportedly higher bond strength of permanent dentin [8,15]. Due to the canal wall preparation and also by moving away from the pulp dentin, intertubular dentin is increased which subsequently improves the bond strength as described earlier.

**Failure modes:**

Studies have shown that failure modes in primary enamel and dentin are usually of adhesive and mixed types [9]. Based on our results, failure modes were significantly different between the total etch and two step self-etch systems; which is in contrast to the findings of Shimadan et al. They found no significant difference in failure modes either between the two systems of total etch and self-etch bonding or between primary and permanent teeth [7]. In their study, failure modes between the one-step and two-step self-etch adhesives and

also between total etch and one-step self-etch were not significantly different.

In two-step Clearfil SE self-etch bond, all failure modes were mixed; which may indicate that this bonding system forms a homogenous hybrid layer that better disseminates the stress in the adhesive area [2]. The greater dispersion of failure modes in the 5<sup>th</sup> generation bonding agents compared to others may indicate their higher technical sensitivity.

Comparable bond strength of self-etch and total etch systems may be explained by the complete penetration of monomer due to the incorporation of MDP. These findings have also been confirmed by several other studies in primary teeth [3, 6, 8, 12,17]. All three bonding systems in our study contained fillers and a water-based solvent. The two self-etch systems also contained MDP monomer. Use of all three systems is recommended for composite posts in primary anterior teeth. However, due to high technical sensitivity of the total etch system (selection of etching time, multi-step etch and rinse system, difficult drying), we recommend using two- or single-step self-etch systems particularly for uncooperative children. The standard deviation of Single Bond 2 bond strength in Table 2 further confirms the technical sensitivity of 5<sup>th</sup> generation bonding agents. Considering this drawback, self-etch systems are superior due to the elimination of this step [14]. Among self-etch systems, single-step bonding agents are superior to two-step systems due to their easy application.

We tried our best to match the conditions of our in-vitro study to the clinical setting. Clinical trials with long-term follow-up are required to assess the durability of the bonds over time.

**CONCLUSION**

In light of the limitations of in-vitro studies, this study showed that all three bonding systems (total etch, one-step self-etch and two-step self-etch) can be used for bonding of composite posts to intracanal dentin of PAT.



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